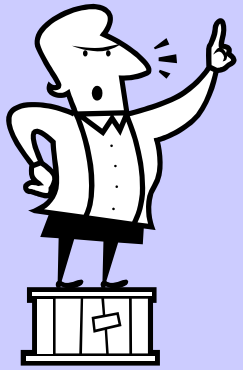
Eurachem

CITAC

Co-Operation on International Traceability in Analytical Chemistry

Guide: Traceability in Chemical Measurements



Overview

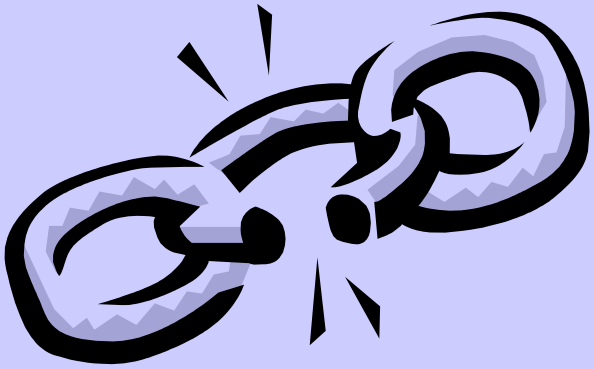
- **Why traceability is important**
- **Principles of Traceability**
- **Application of these principles to chemical measurements**
- **Establishing Traceability**

Traceability

- **“Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards through an unbroken chain of comparisons all having stated uncertainties” (VIM 1993)**

Views of Traceability in Chemical Measurement

A broken chain



A Ball and Chain

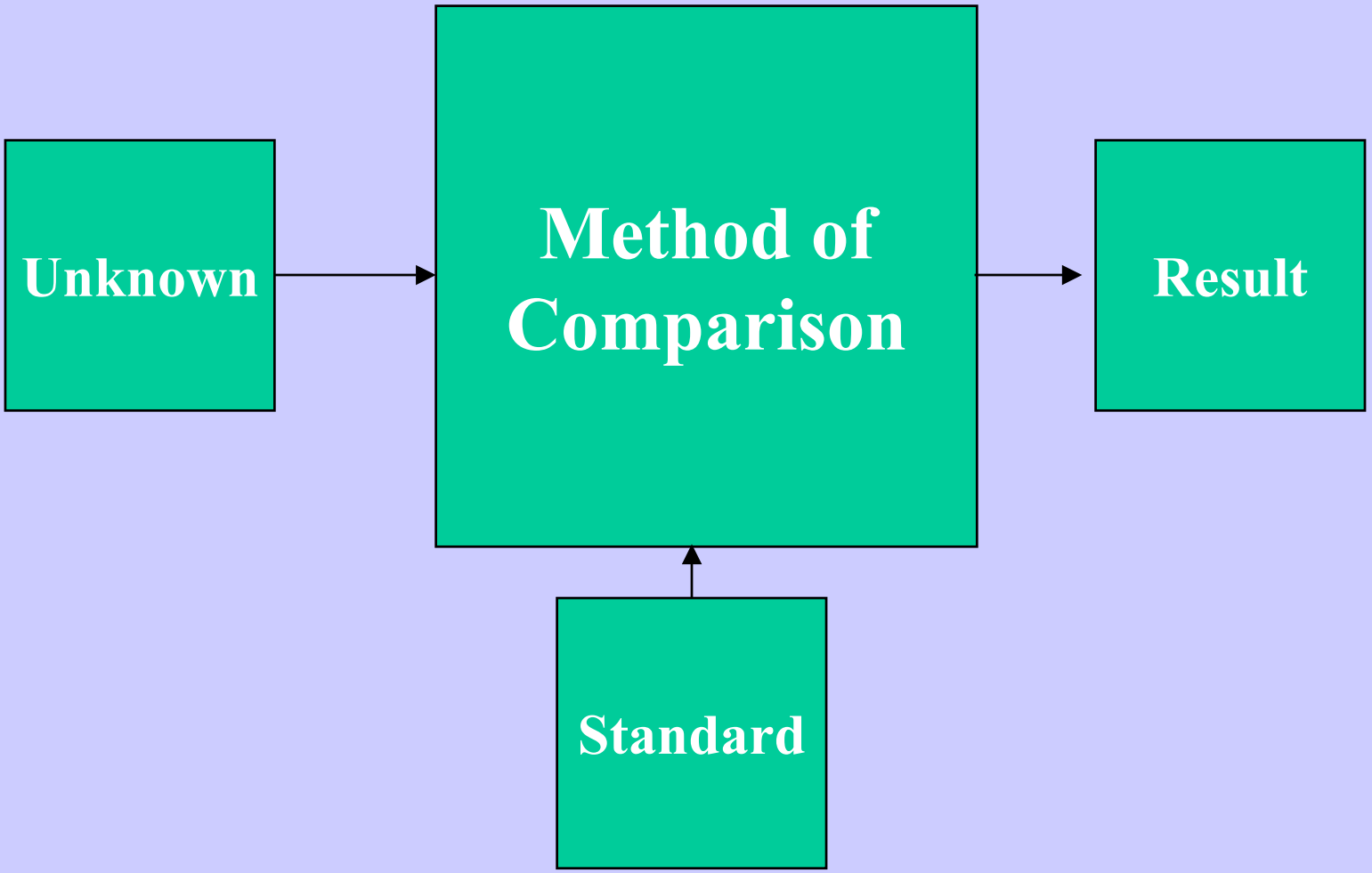
Traceability

- **Traceability is a fundamental property of a result**
- **Not an optional extra**
- **Not just an additional accreditation burden**
- **It enables results to be compared**

Principles Of Measurement

- **Value of the result is obtained from a comparison with the value of a standard**
  e.g. measurement of mass
- **Value of the result is traceable to the value of the standard**
- **Uncertainty on the result is the uncertainty on this comparison plus the uncertainty on the standard**

Principles Of Measurement



Principles Of Traceability

Louisiana Cake Recipe

BETTY RINAUDO'S RUM CAKE

1/2 cup chopped pecans
1/2 cup water
1 box yellow butter cake mix
1 /2 cup light rum
1 small box instant vanilla pudding
4 eggs
1/2 cup oil
1 stick butter
1 cup sugar
1 tbsp. rum
1 tbsp. water

BETTY RINAUDO'S RUM CAKE

1/2 cup (75g) chopped pecans
1/2 cup (125ml) water
1(250g) box yellow butter cake mix
1 /2 cup (125ml) light rum
1 (75g) small box instant vanilla pudding
4 eggs
1/2 cup (125ml) oil
1 stick (150g) butter
1 cup (175g) sugar
1 tbsp.(15ml) rum
1 tbsp.(15ml) water

BETTY RINAUDO'S RUM CAKE

Prepare a 20 oz. bundt pan and sprinkle nuts in bottom.

Combine 1 /2 cup water, cake mix,1 /2 cup rum, pudding mix, eggs and oil. Mix two to three minutes.

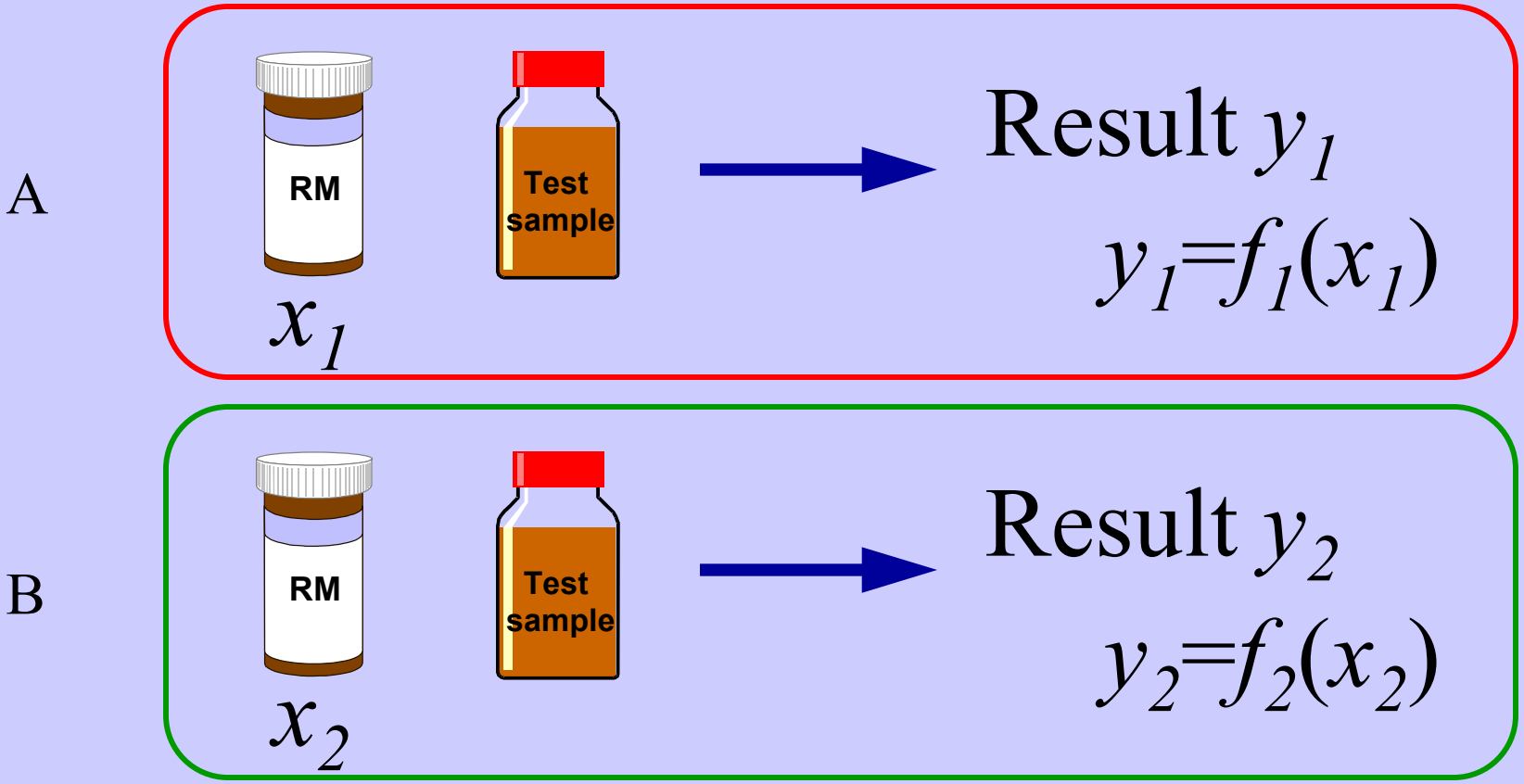
Pour batter into pan and bake at 350 degrees for 50 to 60 minutes.

For glaze, mix 1 stick butter, sugar, 1 tbsp. rum and 1 tbsp. water, then boil about2 minutes.

Pour most of glaze over hot cake and let stand in pan for 30 minutes.

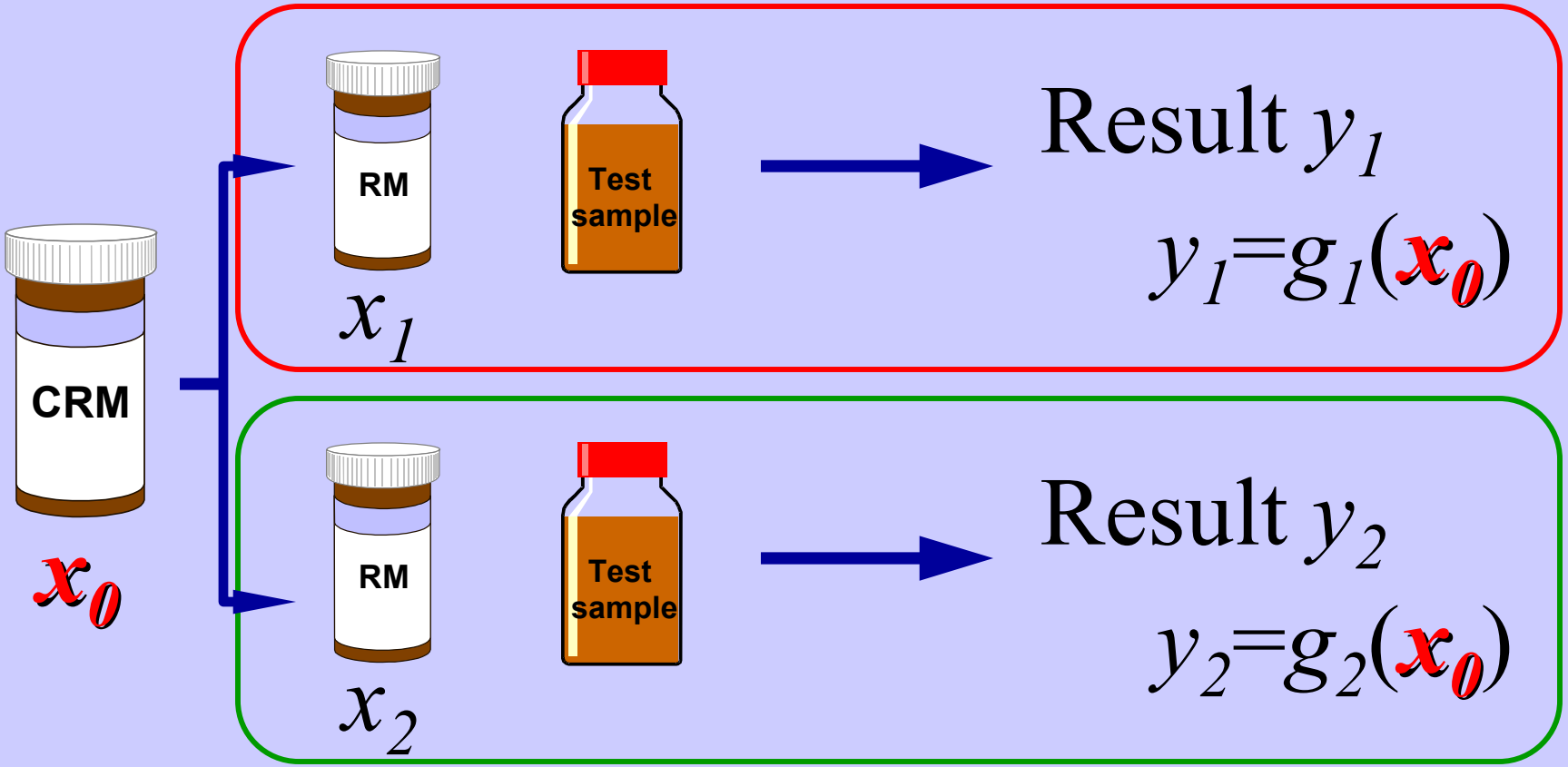
Remove cake from pan and pour remaining glaze over top of cake.

Principles of Traceability



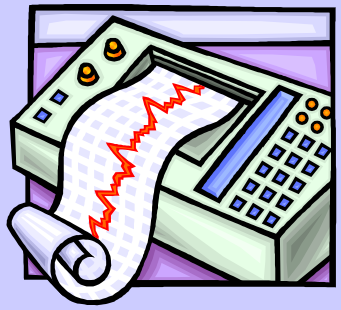
Relationship between y_1 and y_2?

Principles of Traceability



Application to chemical measurements

- **Method development establishes an optimised procedure – Recipe!**
- **Validation demonstrates that this procedure is sufficiently complete for the purpose in hand**
- **Traceability or control has to be established for each parameter specified in the procedure**
- **Traceability is established by calibration using an appropriate measurement standards.**



Application to chemical measurements

A measurement result arises from an equation

$$y = f(x_1, x_2 \ldots \quad x_m)\Big|_{x_{m+1}, x_{m+2} \ldots \quad x_n}$$

- **which is assumed to hold, under certain conditions**
- **value of y is traceable to $x_1, x_2, \ldots x_n$**

Thus, if

$$y = f(x_1, x_2 \ldots \quad x_m)\Big|_{x_{m+1},\, x_{m+2} \ldots \quad x_n}$$

- **The sole requirement for *y* to be fully traceable* is that $x_1 \ldots x_n$ are traceable or defined values**
- **Calibration for $x_1 \ldots x_n$ is sufficient**

**other than MU requirements*

Establishing Traceability

- Specifying the measurand and the acceptable uncertainty
- Choosing a suitable method of estimating the value - that is, a measurement procedure with associated calculation - an equation - and measurement conditions
- Demonstrating, through validation, that the calculation and measurement conditions include all the “influence quantities” that significantly affect the result, or the value assigned to a standard.
- Identifying the relative importance of each influence quantity
- Choosing and applying appropriate reference standards
- Estimating the uncertainty

Choosing reference standards - calibration

- **Physical calibrations are well established**
- **Chemical calibrations can be established in the same way**
  - **pure CRM or matrix calibrant**

  ***OR* (for example)**

  - **using a well-characterised pure material**
- **Uncertainty must be appropriate**

Conclusions

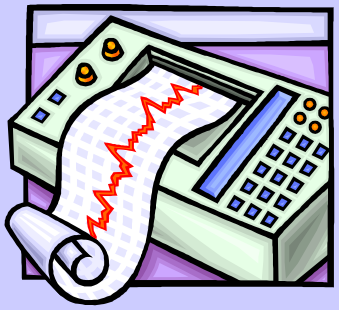
How does this fit in with the definition?

Traceability

- **"Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards through an unbroken chain of comparisons all having stated uncertainties" (VIM 1993)**

Conclusions

- **Method development establishes an optimised procedure – Recipe!**
- **Validation demonstrates that this procedure is sufficiently complete for the purpose in hand**
- **Traceability or control has to be established for each parameter specified in the procedure**
- **Traceability is established by calibration using an appropriate measurement standards.**
- **These standards are traceable to higher level standards. Thus the result is traceable to the values of these higher level standards**



Uncerainty

Live **with** uncertainty

not

in uncertainty

Application to chemical measurements

Assumptions

- An effective quality system is in place
  - Trained Staff
  - Relevant analytical QC and QA in place
  - Documented methods
- Except for
  - Technical elements of traceability

Conclusions

If validation has shown that

$$y = f(x_1, x_2 \ldots \quad x_m) \quad x_{m+1}, x_{m+2} \ldots \quad x_n$$

- **Then the value of y, the result, is traceable to the values of $x_1 \ldots x_n$**

- **The values of $x_1 \ldots x_n$ are traceable to the values of the standards used in calibration**

- **Thus the result is traceable to the values of these standards, which in turn are traceable to higher level standards**

Specifying the measurand

- Identity of the analyte
- Implied measurement conditions
- Recovery correction
- Specification in terms of a method

Choosing a suitable method

If we assume

$$y = f(x_1, x_2 \ldots \quad x_m)\Big|_{x_{m+1}, x_{m+2} \ldots \quad x_n}$$

- **The assumption(s) involved must be tested and shown to hold**
- **This is an essential part of method validation**
- **Validation is crucial to establishing traceability**

Validation

- **Unbiased**
  - **Test with relevant independent CRM, spiking, intercomparisons, …..**
- **Linear (test *via* linearity checks)**
- **Complete - No other effects**
  - **Robustness/ruggedness; Interlaboratory studies …**

Estimating the uncertainty

- **Necessary for the result**
- **Covered in uncertainty Guide**
- **The uncertainties from the measurement procedure and on the values of the standards must be included**